Spray Analysis Report

Customer Information: Rieke/Tommy Liu for Henkel
Sample Information/ID: Renuzit sample set
Analyst: KButz
Instrument: MAL1011117
Quote/Invoice No: RIE-1

Summary:

Seven trigger samples of odor fresheners were submitted to Spray Analytics for droplet size distribution testing to be carried out with the Malvern Spraytec under several conditions. The sample set included the following:

Renuzit Pure Breeze Lavender	Samples 1 and 4
Renuzit Fresh Lavender	Samples 2 and 5
Renuzit Sensitive Scents	Samples 3 and 6
Febreeze	Sample 7

The devices were investigated for droplet size distribution and the final testing was performed in three separate test methods as follows:

Direct spray through test to achieve full spray droplet size distribution

Spray measurement time test to observe the airborne time at distance and height

Bounce back spray measurement to observe spray behavior off of surface

This report will contain three specific sections detailing the methodology and results of each set of testing that was performed. Final conclusions will be detailed at the end of the report.

Section 1: Direct Measurement – Straight Through Testing

Methodology Used:

Each sample was primed prior to testing to ensure full spray was measured.

Spray was positioned six inches from the measurement beam with the center of the beam at the center of the spray plume.

Measurement was made for 1s at 500Hz to ensure full spray capture for all samples

Full spray was averaged to obtain results

Each sample tested in triplicate

Results:

The final averaged data is presented in the attached individual DSD reports. Below is a summary table of the droplet size testing.

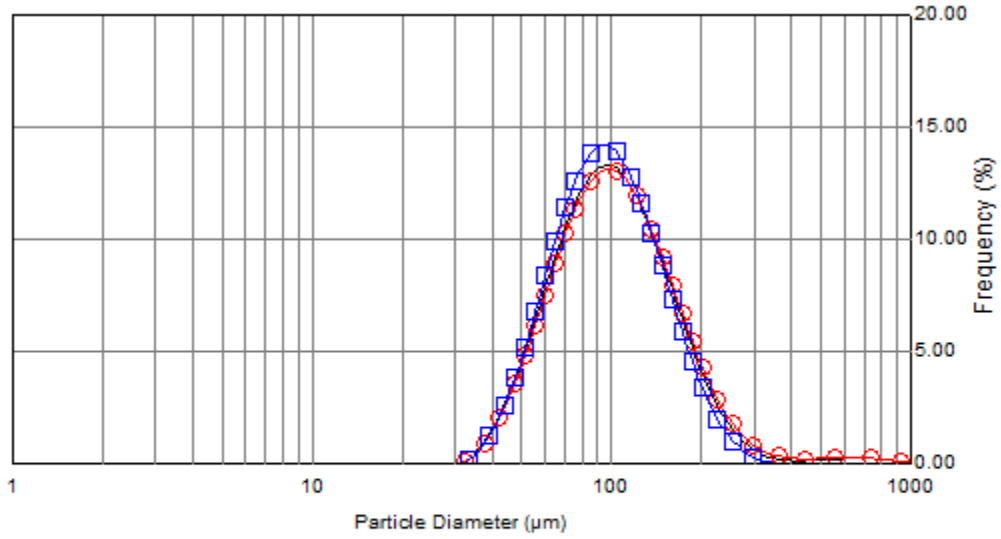
Sample	Dv(10)	Dv(50)	Dv(90)	Dv(100)	%V < 10µ	%V < 7.7µ	%V < 1.9µ
Sample 1 Pure Breeze 1	57.013	99.323	180.787	997.211	0.000	0.000	0.000
Sample 1 Pure Breeze 2	57.780	101.095	187.080	997.135	0.000	0.000	0.000
Sample 1 Pure Breeze 3	56.918	95.996	165.743	853.135	0.000	0.000	0.000
Average	57.237	98.805	177.870	949.160	0.000	0.000	0.000
Sample 2 Fresh Lavender 1	50.652	97.674	210.420	1008.322	0.000	0.000	0.000
Sample 2 Fresh Lavender 2	49.698	93.486	185.266	994.983	0.072	0.072	0.072
Sample 2 Fresh Lavender 3	52.578	102.545	235.439	996.542	0.000	0.000	0.000
Average	50.976	97.902	210.375	999.949	0.024	0.024	0.024
Sample 3 Sensitive Scents 1	61.064	109.385	209.752	994.855	0.000	0.000	0.000
Sample 3 Sensitive Scents 2	60.344	108.257	209.983	996.940	0.000	0.000	0.000
Sample 3 Sensitive Scents 3	58.782	108.026	217.132	996.933	0.000	0.000	0.000
Average	60.064	108.556	212.289	996.242	0.000	0.000	0.000
Sample 4 Pure Breeze 1	50.663	90.313	164.848	643.931	0.000	0.000	0.000
Sample 4 Pure Breeze 2	48.789	88.061	164.355	995.296	0.000	0.000	0.000
Sample 4 Pure Breeze 3	48.141	86.129	158.168	996.898	0.000	0.000	0.000
Average	49.198	88.168	162.457	878.708	0.000	0.000	0.000
Sample 5 Fresh Lavender 1	52.840	96.322	189.586	1009.726	0.000	0.000	0.000
Sample 5 Fresh Lavender 2	50.403	92.113	183.369	1011.696	0.000	0.000	0.000
Sample 5 Fresh Lavender 3	48.318	91.110	188.916	996.215	0.072	0.072	0.072
Average	50.520	93.182	187.290	1005.879	0.024	0.024	0.024

Results (cont):

Sample	Dv(10)	Dv(50)	Dv(90)	Dv(100)	%V < 10µ	%V < 7.7µ	%V < 1.9µ
Sample 6 Sensitive Scents 1	57.651	99.738	180.356	997.411	0.000	0.000	0.000
Sample 6 Sensitive Scents 2	54.575	95.359	180.373	1009.824	0.000	0.000	0.000
Sample 6 Sensitive Scents 3	52.917	93.088	177.993	1010.196	0.000	0.000	0.000
Average	55.048	96.062	179.574	1005.811	0.000	0.000	0.000
Sample 7 Febreeze 1	53.395	142.664	483.654	1007.069	0.246	0.094	0.000
Sample 7 Febreeze 2	47.343	117.897	391.067	997.284	0.338	0.172	0.000
Sample 7 Febreeze 3	48.531	130.631	460.502	1003.303	0.330	0.170	0.000
Average	49.756	130.397	445.074	1002.552	0.305	0.145	0.000

Analysis and Interpretation of Direct Measurements:

Overlay of repeat measurements example:

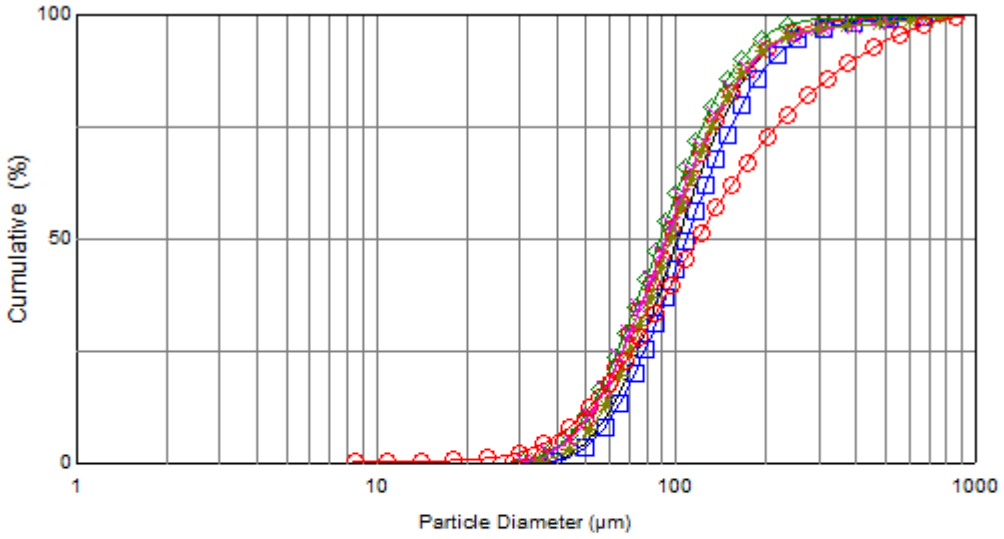


	File-Time	Dx(10)e	Dx(50)	Dx(90)
[V]	Sample 1 Pure Breeze 1 1	57.01	99.32	180.79
[V]	Sample 1 Pure Breeze 1 2	57.78	101.10	187.08
[V]	Sample 1 Pure Breeze 1 3	56.92	96.00	165.74

Above is the frequency representation for the triplicate analysis of sample 1. It shows a high level of consistency across the three sprays that were measured. This is indicative of a pump that is delivering a consistent spray for each time and is priming properly each trigger. This behavior was observed for all seven samples included in the testing.

Analysis and Interpretation of Direct Measurements (cont):

Overlay of Seven Samples



	File-Time	Dx(10)e	Dx(50)	Dx(90)
[V]	Sample 1 Pure Breeze 1 2	57.78	101.10	187.08
[V]	Sample 2 Fresh Lavender 1 2	49.70	93.49	185.27
[V]	Sample 3 Sensitive Scents 2	60.34	108.26	209.98
[V]	Sample 4 Pure Breeze 1 2	48.79	88.06	164.35
[V]	Sample 5 Fresh Lavender 1 2	50.40	92.11	183.37
[V]	Sample 6 Sensitive Scents 2	54.58	95.36	180.37
[V]	Sample 7 Febreeze 1 2	47.34	117.90	391.07

Above is the cumulative trace for all seven samples tested with the second replicate. In the cumulative representation, the S curves farthest to the left on the graph represent the smallest sizes while the trace farthest to the right indicates the largest droplet size.

Above, there is good consistency across all Renuzit samples (1-6) as a thick S curve is observed. The steepness of this thick S curve indicates the spray sizes output are fairly narrow. The Dx(50) value (median droplet size) varies between 88um and 108um which is a small difference given six samples tested under different conditions. As well, the edges of the distributions (Dx(10) and Dx(90) – 10^{th} and 90^{th} percentiles) are consistent indicating the samples all behave in a similar manner.

The Febreeze sample is the red dashed trace that is seen farthest to the right at larger droplet sizes. While the atomization of the fine droplets is similar as marked by the trace's inclusion into the thick S curve on the left side of the trace, the Febreeze has larger droplets output on the large side of the distribution. This is also seen by the Dx(90) value being much larger than the Renuzit samples.

Overall, only the Febreeze sample behaved differently and this was only on the large side of the distribution. Febreeze is outputting a larger max size. On the fine side of the distribution, there was little difference between all seven samples.

Maximum Size, Median Size and Inhalation Information:

Maximum Size:

The maximum size reported in each spray was measured as the Dx(100) in the prior table. This indicates the 100^{th} percentile of the spray. This value is a calculation of the largest droplet output by each sample and generally will have more variability than values in the center of the droplet size distribution.

Median Size:

The median size reported in each spray is noted by the Dx(50) in the tables above. This value was fairly consistent across all seven samples measured. The Febreeze sample showed a slightly larger value that is caused primarily by the excess large droplets involved in the spray which moves the median droplet size larger as well.

Inhalation Information:

In most studies, the percentage below 10um is used to quantify the amount of inhalation risk. In this study, 7.7um and 1.9um were additionally quantified for further information. None of the samples involved registered above 0.5% at any of the three points recorded. The Febreeze sample showed the highest quantities of droplets but still were found to be well under 1% of the volume of the spray.

Section 2: Timed Measurement

Methodology Used:

Each sample was primed prior to testing to ensure full spray was measured.

Spray was positioned twelve inches from the measurement beam with the center of the beam at the center of the spray plume.

Only a portion of the spray was allowed to travel through the beam to contain the spray in the measurement zone.

Measurement was made for 5s at 100Hz to allow full spray capture and fall for all samples

Full spray was averaged to obtain results

Each sample tested in triplicate

Results:

Sample	Time Measured
Sample 1 Pure Breeze 1	3.930
Sample 1 Pure Breeze 2	3.440
Sample 1 Pure Breeze 3	3.590
Average	3.653
Sample 2 Fresh Lavender 1	3.590
Sample 2 Fresh Lavender 2	4.410
Sample 2 Fresh Lavender 3	3.560
Average	3.853
Sample 3 Sensitive Scents 1	2.590
Sample 3 Sensitive Scents 2	2.060
Sample 3 Sensitive Scents 3	1.560
Average	2.070
Sample 4 Pure Breeze 1	2.920
Sample 4 Pure Breeze 2	2.690
Sample 4 Pure Breeze 3	4.300
Average	3.303

Sample	Time Measured
Sample 5 Fresh Lavender 1	4.990
Sample 5 Fresh Lavender 2	4.720
Sample 5 Fresh Lavender 3	4.880
Average	4.863
Sample 6 Sensitive Scents 1	3.640
Sample 6 Sensitive Scents 2	3.530
Sample 6 Sensitive Scents 3	3.110
Average	3.427
Sample 7 Febreeze 1	4.420
Sample 7 Febreeze 2	3.730
Sample 7 Febreeze 3	3.740
Average	3.963

Analysis and Interpretation:

No droplet size data is reported above as the goal of the experiment was to measure the length of time the spray is present. Measurements with the Malvern Spraytec are allowed to be set up to measure dilute droplet concentrations. The threshold for measurement for this study was set up to be very low so as to measure very small amounts of droplets.

The measurements were set up to be made for 5s. The Malvern Spraytec will only report results for the time the concentration of droplets is above the threshold set. Therefore, the values reported are from the data points that were recorded. The values are all below 5s as the spray was not present at the testing distance for the full 5s. As the testing was performed at 100Hz, the measured times reflect values where measurement was performed as the droplet concentration was above the threshold to make a droplet size measurement. Using this strategy, we can use the measurement to provide the time detail in the table above.

In this testing, the measurements were made strictly at the same height as the bottle. Therfore, material falling from gravity over time will not be measured. The smaller droplets that are experiencing evaporation and initial spray momentum are prone to linger in the measurement zone and be measured over time.

12 inches was selected for measurement after observing the spray. Seeing a billowing of the spray plume at a distance is generally reflective of the end of the true spray plume as the initial momentum of the droplets has been nearly stopped.

In the data table, it can be said that there is no distinct pattern of spray length. As this testing is less controlled (spraying in an open space from a longer distance) the room conditions are more critical to the data than some other testing methods. However, this testing is done in conditions most similar to customer conditions. The primary difference in this situation is the spray is left to fully evolve as it is sprayed through the beam as opposed to onto a surface. For that reason, the times reported above are likely to be more of a worst case scenario situation of a user spraying the product into an open ended environment as opposed to on a specific target.

While there may be some trends available in the timed data above, caution in suggesting performance of any one sample being different than another sample would be challenging to prove statistically given the limited sample set testing. For the purposes of exposure, the data provided should be sufficient along with the prior droplet size data provided.

Section 3: Spray Bounce Back Measurement

Methodology Used:

Each sample was primed prior to testing to ensure full spray was measured.

Spray was positioned six inches above the measurement beam directly above the beam.

The surface sprayed onto was a hard plastic to facilitate as much bounce back as possible to emphasize any issue that could be present. The surface was positioned six inches from sprayer.

The samples were triggered into the surface and the bounce off of the surface was required to trigger a measurement.

Measurement was made for 3s at 100Hz to allow full bounce back spray to be measured

Full spray was averaged to obtain results

Results:

Sample 1 Pure Breeze	**Trans**	**Dv(10)**	**Dv(50)**	**Dv(90)**	**%V < 10µ**	**%V < 7.7µ**	**%V < 1.9µ**
Bounce Back	99.836	38.993	112.092	692.281	0.319	0.014	0.000
Straight Through Average	79.403	57.237	98.805	177.870	0.000	0.000	0.000
Sample 2 Fresh Lavender							
Bounce Back	99.786	32.054	109.255	198.085	2.879	2.195	0.303
Straight Through Average	77.953	50.976	97.902	210.375	0.024	0.024	0.024
Sample 3 Sensitive Scents							
Bounce Back	99.720	34.813	458.686	766.139	1.651	1.322	0.081
Straight Through Average	82.281	60.064	108.556	212.289	0.000	0.000	0.000
Sample 4 Pure Breeze							
Bounce Back	99.664	18.206	37.429	82.778	5.113	5.113	0.003
Straight Through Average	75.590	49.198	88.168	162.457	0.000	0.000	0.000
Sample 5 Fresh Lavender							
Bounce Back	99.864	27.119	50.039	657.125	0.000	0.000	0.000
Straight Through Average	76.312	50.520	93.182	187.290	0.024	0.024	0.024
Sample 6 Sensitive Scents							
Bounce Back	99.593	23.856	66.059	185.559	1.342	0.947	0.021
Straight Through Average	76.431	55.048	96.062	179.574	0.000	0.000	0.000
Sample 7 Febreeze							
Bounce Back	99.844	25.908	109.061	669.417	0.000	0.000	0.000
Straight Through Average	79.681	49.756	130.379	445.074	0.305	0.145	0.000

Analysis and Interpretation:

In the table above, the bounce back single measurement is compared to the average from the Direct measurements of the same sample.

The bounce back testing was run as a single sample for each sample because of the dilute nature of the bounce back spray and its inconsistency in occurrence. Often times, a single spray was insufficient in surpassing the threshold required to activate the measurement. For that reason, a single measurement was made for each sample in this study.

The value in the first column is that of Transmission. This refers to the measurement beam and how much of it is transmitted through the sample during the measurement. As the transmission goes down, the amount of material in the beam is greater. For that reason, the transmission value can be used for relative concentration of droplets in the measurement.

In the direct spray testing (straight through), the transmission values observed were in the 80% range for all samples tested indicating the full spray produces this transmission. In the bounce back spray, the transmissions recorded were all in the 99.5% or more range. This suggests that less than 2.5% of the initial spray is bouncing back a distance of 6 inches at a height of 6 inches below the sprayer. This volume of bounced spray can be compared to the full volume of a spray when suggesting exposure risks. The location of the bounce back measurement is likely closer to the surface and off a surface that promotes bounce more than the intended use. These locations were chosen as a possible worst case scenario where data would be able to be measured with the Malvern Spraytec set up to test a low threshold of droplets.

The spray sizes reported above in the bounce back data represent the spray observed after bouncing off the surface. These results are expected to be very inconsistent as they are encountering the surface which changes their direction. In the cases where the droplet size is seen as larger, a few very large droplets are rebounding off the surface.

In term of inhalation, the bounce back readings are found to be generally higher than the direct measurement. This is expected and must be considered along with the concentration of droplets to establish exposure risk. While a higher percentage of the material measured may be found to be at or below a 10um size, it is important to consider the amount of material measured is much less than the direct measurement (approximately 1-2% of initial spray is measured to be bouncing back).

Conclusions:

1. In the direct measurement section, each sample sprayed consistently across the three spray events measured. This indicates a functional trigger that is able to prime the sample repeatedly and consistently and output the same or very similar spray plume repeatedly.
2. The six Renuzit samples all show a very similar droplet size distribution to one another. The Febreeze sample shows a higher top end size in comparison with the Renuzit samples. This means the Febreeze deviates from the other samples only on the large droplet sizes. This is measured most effectively by the Dx(90) value that is reported in the table.
3. None of the seven samples show more than 0.5% of the material out of the spray trigger to be under 10um which is often considered to be the size indicative of an inhalation risk. This suggests minimal inhalation risk.
4. The time measurements were conducted to see how long the spray will linger in the air. In this study, no specific conclusions related to one sample versus another could be definitively determined. All seven samples show some similarity in the amount of time the formulation is lingering and measurable in the beam. The environment the sample is sprayed in will have some impact on these results. The environment the testing was conducted under would be equivalent to the user spraying into an open space with nothing to stop the spray.
5. The amount of material bouncing back at the spray user was deemed to be very small in terms of volume sprayed in total. Worst case scenario appears 2.5% or less of each sample is bouncing back. While a higher percentage of the droplets measured were in the inhalation range for size, it needs to be reiterated that the amount of material bouncing back was very low. As well, the location of the sprayer and measurement beam along with the surface sprayed onto was meant to maximize bounce back to show a worst case scenario for the product. The small amount of material at the low sizes suggests little inhalation risk.

Understanding Your Droplet Size Study

Understanding the Report:

The data report following includes several size distribution values. The most commonly used values are defined below:

Trans – Transmission of measurement laser beam through the sample. This can be used to qualitiatively evaluate the amount of spray present in one measurement vs another.

Dv(10) – Indicative of the 10^{th} percentile by volume of the spray.

Dv(50) – Indicative of the 50^{th} percentile by volume of the spray. The median droplet size

Dv(90) – Indication of the 90^{th} percentile by volume of the spray

Dv(100) - Indication of the 90^{th} percentile by volume of the spray

Span – A calculation of breadth of distribution noted by (Dv(90)-Dv(10))/Dv(50)

% Volume < (um) – Measurement of the sample below a specific droplet size.

D[4,3] – Volume weighted mean of a spray.

D[3,2] – Surface weighted mean of a spray.

If you have any questions/concerns about the data presented in this report, please contact us to discuss the results in further detail.

Thank You

Kyle Butz